UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement and Compliance Assurance
Office of Criminal Enforcement, Forensics and Training

NEICVP0843E01

ENFORCEMENT CONFIDENTIAL

LABORATORY REPORT

Dalton, Georgia, Water Supply Analyses
Dalton, Georgia
NEIC Project Number: VP0843

July 2009

Project Leader:

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Prepared for:

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EXECUTIVE SUMMARY

INTRODUCTION

The U.S. Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) provided laboratory assistance to EPA Region 4, Water Protection Division, in an investigation of the possible presence of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS), as well as other fluorinated compounds, in north Georgia public water systems. In January 2009, EPA issued Provisional Health Advisories for PFOA and PFOS. The provisional health advisory concentrations are 400 ng/L (nanograms per liter or parts per trillion) for PFOA and 200 ng/L for PFOS. This report provides analytical results for PFOA and PFOS for samples collected by EPA Region 4 personnel March 31 to April 1, 2009. Results for other analytes will be provided in a subsequent report.

In the laboratory analysis employed, both PFOA and PFOS are detected as anions. This report presents results for PFOA as the acid form and for PFOS as the anion form. For PFOA, acid-form results can be converted to anion-form results by multiplying by 0.9976. Anion- and acid-form results are indistinguishable at the accuracy of the results presented in this report. The samples were analyzed using liquid chromatography/tandem mass spectrometry (LC/MS/MS). All analyses performed at the NEIC laboratory were conducted by NEIC personnel in accordance with the NEIC quality system.

SUMMARY OF FINDINGS

- The NEIC laboratory identified both PFOA and PFOS in samples from 2 of 15 stations (R1F and R1R1). Both samples contained PFOA and PFOS at levels less than the provisional health advisory concentrations (400 ng/L for PFOA and 200 ng/L for PFOS).
- PFOA and PFOS were not detected in any of the trip blank water samples.

LABORATORY ACTIVITIES

SAMPLE INFORMATION

Larry Strattan received 15 water samples and two trip blanks in a total of 70 containers, which were handled in accordance with the NEIC procedure, *Evidence Management* (NEICPROC/00-059R2). One trip blank was preserved with sodium thiosulfate, and the other was not, in order to serve as blanks for finished and raw water, respectively. Each trip blank consisted of five containers, two 500-milliliter (mL) containers and three 250-mL containers. Sample portions from 500-mL containers were analyzed. A copy of the chain-of-custody record is attached as **Appendix A**.

ANALYTICAL PROCEDURES

The procedures used to analyze the water samples for PFOA and PFOS were derived primarily from articles[1][2] published in scientific literature. Approximately 100-mL samples were analyzed using solid phase extraction (SPE) and liquid chromatography/tandem mass spectrometry incorporating isotope dilution quantitation. Isotope dilution was performed by adding $^{13}C_4$-labeled PFOA and PFOS (MPFOA and MPFOS, respectively) to each sample at concentrations of 500 ng/L and 477.5 ng/L before extraction, assuming a 100-mL sample volume. MPFOA and MPFOS were used as internal standards to calculate the concentrations of the unlabeled compounds present in extracts. The recoveries of MPFOA and MPFOS were evaluated to ensure that there were no values below 50 percent, which would indicate that reporting limits should be adjusted for individual samples. There were no such instances.

Quality assurance steps included the analysis of (1) instrument and preparation blanks, (2) replicate samples, (3) matrix spikes of PFOA and PFOS, (4) calibration standards intermixed with samples, and (5) alternate-source standards. PFOA was identified in instrument and preparation blanks, and the PFOA reporting limit (approximately 80 ng/L) was chosen to be above the concentrations observed in blanks. PFOS was not identified in blanks. The PFOS reporting limit (approximately 60 ng/L) was based on the reference standard concentration necessary to result in a chromatographic response signal-to-noise ratio of at least three at both the quantitation ion (mass-to-charge ratio, m/z, 499 → 80) and confirmation ion (m/z 499 →99).

[1] Moody, C.A.; Kwan, W.C.; Martin, J.W.; Muir, D.C.G.; Mabury, S.A. *Anal. Chem.* **2001**, *73*, 2200-2206.
[2] Risha, Karen; Flaherty, John, *et al*, *Anal. Chem.* **2005**, *77*, 1503-1508.

ANALYTICAL RESULTS

Table 1 shows the results of these analyses. The table provides the station identification from the chain-of-custody record, the bottle number analyzed, and the results for PFOA and PFOS. Qualitative results are expressed as positive or negative. Quantitative results or reporting limits have concentration units of ng/L. Samples from stations R1F and R1R1 were analyzed multiple times, and the individual results, as well as the mean, standard deviation, relative standard deviation, and 95 percent confidence intervals, are shown in Tables 1 and 2.

The bottle 53 replicate from station R1R1 with a PFOA result of 75 ng/L is just below the reporting limit of 80 ng/L. The result is reported as a positive identification because the other two replicates indicated the presence of PFOA, and chromatographic responses for the precursor, quantitation, and confirmation ions were present at the expected retention time.

Table 3 shows results for three spikes of unlabeled PFOA and PFOS into samples. The spiked concentration is the concentration of compound in the sample portion before extraction. Neither PFOA nor PFOS was detected in the unspiked portions of these samples.

Table 1. ANALYTICAL RESULTS
Dalton, Georgia, Water Supply Analyses
Dalton, Georgia

Station	Bottle Number	PFOA (ng/L)	PFOS (ng/L)
C1F	1	Negative <[1]79 Negative <85	Negative <59 Negative <64
C1R	8	Negative <90	Negative <67
C2F	9	Negative <79	Negative <59
C2R	16	Negative <86	Negative <64
D1F	17	Negative <83	Negative <62
D1R	24	Negative <81	Negative <61
D2F	25	Negative <84	Negative <63
D2R	30	Negative <81	Negative <61
D3F	33	Negative <91	Negative <68
D3R	37	Negative <87	Negative <66
F1F	43	Negative <86	Negative <65
F1R	47	Negative <82	Negative <62
R1F[2]	49	Positive, < 130	Positive 88, 146, 109 mean = 114 s = 29 RSD = 25% 95% confidence interval: 40 to 190
R1R1[2]	53	Positive, < 110	Positive 99, 76, 90 mean = 88 s = 11 RSD = 13% 95% confidence interval: 60 to 117
R1R2	57	Negative <82	Negative <61
Trip Blank	61	Negative <81	Negative <61
Trip Blank	62	Negative <83	Negative <62
Preserved Trip Blank	66	Negative <78	Negative <57
Preserved Trip Blank	67	Negative <84	Negative <63

[1] < Indicates not present above the indicated reporting limit; reporting limits vary due to the sample volume extracted.

[2] Samples showing a mean, standard deviation (s), and relative standard deviation (RSD) were analyzed in triplicate; results for all three analyses are reported.

Table 2. PFOA SUMMARY STATISTICS
Dalton, Georgia, Water Supply Analyses
Dalton, Georgia

Station	Bottle Number	PFOA (ng/L)
R1F[2]	49	87, 112, 88
		mean = 95
		s = 14
		RSD = 15%
		95% confidence interval: 60 to 130
R1R1[2]	53	75, 91, 82
		mean = 83
		s = 8.0
		RSD = 9.8 %
		95% confidence interval: 63 to 103

Table 3. MATRIX SPIKE RESULTS
Dalton, Georgia, Water Supply Analyses
Dalton, Georgia

Analyte	Spiked Concentration (ng/L)	Recovery (%)
Station C1R – 1		
PFOA	465	79.8
PFOS	370	77.4
Station F1F – 43		
PFOA	196	87.4
PFOS	156	91.2
PFOA	477	79.9
PFOS	379	70.5

APPENDIX A

CHAIN-OF-CUSTODY RECORDS
Project No. VP0843
(3 pages)

VT0843

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region: 4	Date Shipped: 4/1/2009	Chain of Custody Record	Sampler Signature: [signature]
Project Code: 09-0322	Carrier Name: FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code: 09-0322	Airbill:	1 [signature] 4-1-09/1400	[signature] 4/2/09 08:38
CERCLIS ID:	Shipped to: EPA/NEIC Denver Federal Center Bldg 25 Entrance 52 Denver CO 80225 (303) 462-9106	2.	
Spill ID:		3.	
Site Name/State: North Georgia Public Water Supply PFC		4.	
Project Leader: Mike Neill			
Action:			
Sampling Co: PL-SESD			

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
C1F	Potable Water/ Marty Allen	L/G	PFCs (35)	1 (Na2S2O3), 2 (Na2S2O3), 3 (Na2S2O3), 4 (Na2S2O3) (4)	C1F	S: 3/30/2009 16:07	1-4 1=500mL (underlined number)
C1R	Potable Water/ Marty Allen	L/G	PFCs (35)	5 (Not preserved), 6 (Not preserved), 7 (Not preserved), 8 (Not preserved) (4)	C1R	S: 3/30/2009 16:05 5-8	-
C2F	Potable Water/ Marty Allen	L/G	PFCs (35)	10 (Na2S2O3), 11 (Na2S2O3), 12 (Na2S2O3), 9 (Na2S2O3) (4)	C2F	S: 3/30/2009 16:45 9-12	-
C2R	Potable Water/ Marty Allen	L/G	PFCs (35)	13 (Not preserved), 14 (Not preserved), 15 (Not preserved), 16 (Not preserved) (4)	C2R	S: 3/30/2009 16:40 13-16	-
D1F	Potable Water/ Marty Allen	L/G	PFCs (35)	17 (Na2S2O3), 18 (Na2S2O3), 19 (Na2S2O3), 20 (Na2S2O3) (4)	D1F	S: 3/30/2009 13:17 17-20	-
D1R	Potable Water/ Marty Allen	L/G	PFCs (35)	21 (Not preserved), 22 (Not preserved), 23 (Not preserved), 24 (Not preserved) (4)	D1R	S: 3/30/2009 13:15 21-24	-
D2F	Potable Water/ Marty Allen	L/G	PFCs (35)	25 (Na2S2O3), 26 (Na2S2O3), 27 (Na2S2O3), 28 (Na2S2O3) (4)	D2F	S: 3/30/2009 14:17 25-28	-
D2R	Potable Water/ Marty Allen	L/G	PFCs (35)	29 (Not preserved), 30 (Not preserved), 31 (Not preserved), 32 (Not preserved) (4)	D2R	S: 3/30/2009 14:15 29-32 30=500mL	-

Not confirmed bottle #s 4/15/09

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? yes

PFCs = Perfluorinated Compounds

TR Number: 4-043013577-040109-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

VP0843

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	**Date Shipped:**	4/1/2009	**Chain of Custody Record**	**Sampler Signature:** [signature]
Project Code:	09-0322	**Carrier Name:**	FedEx	**Relinquished By (Date / Time)**	**Received By (Date / Time)**
Account Code:	09-0322	**Airbill:**		1 [signature] 4-1-09/1400	[signature] 4/15/09 0838
CERCLIS ID:		**Shipped to:**	EPA/NEIC Denver Federal Center Bldg 25 Entrance 52 Denver CO 80225 (303) 462-9106	2.	
Spill ID:				3.	
Site Name/State:	North Georgia Public Water Supply PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME				QC Type
D3F	Potable Water/ Marty Allen	L/G	PFCs (35)	33 (Na2S2O3), 34 (Na2S2O3), 35 (Na2S2O3), 36 (Na2S2O3) (4)	D3F	S: 3/30/2009	15:00	33-36		-
D3R	Potable Water/ Marty Allen	L/G	PFCs (35)	37 (Not preserved), 38 (Not preserved), 39 (Not preserved), 40 (Not preserved) (4)	D3R	S: 3/30/2009	14:57	37-40		-
F1F	Potable Water/ Marty Allen	L/G	PFCs (35)	41 (Na2S2O3), 42 (Na2S2O3), 43 (Na2S2O3), 44 (Na2S2O3) (4)	F1F	S: 3/31/2009	10:55	41-44	43	-
F1R	Potable Water/ Marty Allen	L/G	PFCs (35)	45 (Not preserved), 46 (Not preserved), 47 (Not preserved), 48 (Not preserved) (4)	F1R	S: 3/31/2009	10:45	45-48	47	-
Q1	Trip Blank Water/ Marty Allen	L/G	PFCs (35)	61 (Not preserved), 62 (Not preserved), 63 (Not preserved), 64 (Not preserved), 65 (Not preserved) (5)	Q1	S: 3/31/2009	8:15	61-65	61 & 62	QC (Field)
Q2	Preservative Blank/ Marty Allen	L/G	PFCs (35)	66 (Na2S2O3), 67 (Na2S2O3), 68 (Na2S2O3), 69 (Na2S2O3), 70 (Na2S2O3) (5)	Q2	S: 3/31/2009	8:15	66-70	66 & 67	QC (Field)
R1F	Potable Water/ Marty Allen	L/G	PFCs (35)	49 (Na2S2O3), 50 (Na2S2O3), 51 (Na2S2O3), 52 (Na2S2O3) (4)	R1F	S: 3/31/2009	9:38	49-52		-
R1R1	Potable Water/ Marty Allen	L/G	PFCs (35)	53 (Not preserved), 54 (Not preserved), 55 (Not preserved), 56 (Not preserved) (4)	R1R1	S: 3/31/2009	9:35	53-56		-

4 wt. confirmed bottle IIs upon check-in 4/15/09

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? yes
PFCs = Perfluorinated Compounds			

TR Number: **4-043013577-040109-0001**

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VP0843

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	Date Shipped:	4/1/2009	Chain of Custody Record	Sampler Signature: Mark Neill
Project Code:		Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0322 09-0322	Airbill:		1 Mark Neill 4-09/1400	K. Stratton 4/15/09 9:38a
CERCLIS ID:		Shipped to:	EPA/NEIC Denver Federal Center Bldg 25 Entrance 52 Denver CO 80225 (303) 462-9106	2.	
Spill ID:				3.	
Site Name/State:	North Georgia Public Water Supply PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
R1R2	Potable Water/ Marty Allen	L/G	PFCs (35)	57 (Not preserved), 58 (Not preserved), 59 (Not preserved), 60 (Not preserved) (4)	R1R2	S: 3/31/2009 10:00	-

57-60
KWL 4/15/09

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? yes
PFCs = Perfluorinated Compounds			

TR Number: 4-043013577-040109-0001

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